



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
NATIONAL EXPOSURE RESEARCH LABORATORY  
RESEARCH TRIANGLE PARK, NC 27711

OFFICE OF  
RESEARCH AND DEVELOPMENT

November 9, 2017

**MEMORANDUM**

**SUBJECT:** Lab Results of PFAS Concentrations in Water Samples: NC DEQ Report No. 7

**FROM:** Timothy J. Buckley, Director  
Exposure Methods and Measurements Division

**THRU:** Tim Watkins, Acting Director  
National Exposure Research Laboratory

**TO:** Linda Culpepper, Deputy Director  
Division of Water Resources  
North Carolina Department of Environmental Quality

Attached, please find our laboratory report for samples collected under the direction of NC DEQ on October 16, 18, and 19, 2017. Samples were collected from 8 sites along the Cape Fear River including Chemours outfall and finished drinking water sites. This is our 7<sup>th</sup> report in a series of reports intended to support NC DEQ decisions related to industrial discharge of PFAS to the Cape Fear River and surrounding community.

Thank you for providing us with the opportunity to contribute to this effort for the protection of North Carolina public health and the environment. This work is well aligned with EPA's new Strategic Plan that refocuses EPA on its role of supporting the primary implementers of environmental programs—states and tribes “to provide tangible and real environmental results to the American people.”

These results represent the effort and expertise of Drs. Mark Strynar, Andy Lindstrom, James McCord, and Seth Newton in conducting the laboratory analyses. Dr. Myriam Medina-Vera provided invaluable management support and coordination, and Ms. Sania Tong Argao is acknowledged for her support in quality assurance review.

If you have any questions or concerns, do not hesitate to contact me at (919) 541-2454 or email [buckley.timothy@epa.gov](mailto:buckley.timothy@epa.gov). I look forward to our continued work together.

Enclosure

CC: Becky B. Allenbach, USEPA Region 4  
Jeff Morris, USEPA OPPT  
Betsy Behl, USEPA, OW  
Peter Grevatt, USEPA, OW

## Summary of Methods and Results

The results reported here are from sampling that was conducted by NC DEQ staff on October 16, 18, and 19, 2017 and delivered to our laboratory on October 24, 2017. For this report, we limited our analysis to GenX and the five non-targeted analytes (Table 1). We selected these analytes because previous reports have shown them to be of concern. As we have indicated in prior reports, an important limitation to our non-targeted analysis is that these results are considered semi-quantitative. We cannot know the exact concentration because no authentic standards are available for these chemicals. However, we are very confident of the chemical identity based on the high resolution mass spectrometry and knowledge of Chemours' chemical products.

**Table 1. PFAS Analytes Measured**

Short Name	Chemical Name	Formula	CAS no.	Monoisotopic Mass (Da)
<b>Analyte Measured by Targeted Analysis</b>				
GenX	Perfluoro(2-methyl-3-oxahexanoic) acid	C <sub>6</sub> HF <sub>11</sub> O <sub>3</sub>	13252-13-6	329.9750
<b>Analytes Measured by Non-Targeted Analysis</b>				
PFMOAA	(2,2-difluoro-2-(trifluoromethoxy)acetic acid)	C <sub>3</sub> HF <sub>5</sub> O <sub>3</sub>	674-13-5	179.9846
PFO2HxA	perfluoro-3,5-dioxaheptanoic acid	C <sub>4</sub> HF <sub>7</sub> O <sub>4</sub>	39492-88-1	245.9763
PFO3OA	perfluoro-3,5,7-trioxaoctanoic acid	C <sub>5</sub> HF <sub>9</sub> O <sub>5</sub>	39492-89-2	311.9680
PFESA (Nafion) Byproduct 1	Perfluoro-3,6-dioxa-4-methyl-7-octene-1-sulfonic acid	C <sub>7</sub> HF <sub>13</sub> SO <sub>5</sub>	29311-67-9	443.9337
PFESA (Nafion) Byproduct 2	Ethanesulfonic acid, 2-[1-[difluoro(1,2,2,2-tetrafluoroethoxy)methyl]-1,2,2,2-tetrafluoroethoxy]-1,1,2,2-tetrafluoro-	C <sub>7</sub> H <sub>2</sub> F <sub>14</sub> O <sub>5</sub> S	749836-20-2	463.9399

We determined the concentration of GenX against a standard calibration curve derived from an authentic standard using a traditional targeted analysis approach. In contrast, our quantification of the non-targeted analytes is semi-quantitative. Since standards are not available for these analytes, we estimate their concentrations based on the GenX instrument response as shown in the equation below.

$$[NTA] = [GenX] * \frac{NTA_{PA}}{GenX_{PA}}$$

Where: [NTA] is the concentration of the non-targeted analyte (ng/L)  
 [GenX] is the concentration of GenX (ng/L)  
 NTA<sub>PA</sub> is the integrated peak area for the non-targeted analyte  
 GenX<sub>PA</sub> is the integrated peak area for GenX

This method of estimation assumes that the mass spectrometer responds to the non-targeted analyte as if it were GenX. The actual instrument response may be weaker or stronger resulting in an under- or over-estimation of the non-targeted concentration. Our experience with this class of analytes suggests that estimates of this fashion are accurate to within ~10-fold of the estimated value. Additional uncertainty in estimated concentration of non-targeted analytes is introduced for samples where GenX exceeded the calibration curve (because the determination of GenX concentration is more uncertain) and for samples where there is a large discrepancy between the GenX concentration and the non-target analyte (due to scaling). For GenX, laboratory analysis was performed on an ultra-performance liquid chromatography tandem mass spectrometer, and for the non-targeted analytes, analysis was performed on a quadrupole time of flight mass spectrometer. Additional details on the method of analysis can be found in Sun *et al.*, 2016<sup>1</sup> and Strynar *et al.*, 2015<sup>2</sup>.

Results are presented in Table 2. Concentrations varied by sampling location and analyte. Concentrations of GenX exceeded the highest value on our calibration curve (i.e., 250 ng/L) for two samples, and these values have been flagged accordingly. Additionally, GenX was found in Brunswick County finished drinking water at a concentration (305 ng/L) that exceeds the NC DHHS GenX health goal of 140 ng/L.

Quality control samples included a lab blank and multiple solvent blanks. In all cases, none of the analytes were detected in any blanks. The 6-point calibration curve was within +/- 20% of the theoretical concentration for each curve point from 10 to 250 ng/L for GenX and had an  $R^2$  of >0.99.

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<sup>1</sup> Sun M; Arevalo E; Strynar M; Lindstrom A; Richardson M; Kearns B; Pickett A; Smith C; Knappe DRU: Legacy and Emerging Perfluoroalkyl Substances Are Important Drinking Water Contaminants in the Cape Fear River Watershed of North Carolina. Environmental Science & Technology Letters. 2016

<sup>2</sup>Strynar M, Dagnino S, McMahan R, Liang S, Lindstrom A, Andersen E, McMillan L, Thurman M, Ferrer I, Ball C. Identification of Novel Perfluoroalkyl Ether Carboxylic Acids (PFECAs) and Sulfonic Acids (PFESAs) in Natural Waters Using Accurate Mass Time-of-Flight Mass Spectrometry (TOFMS). Environ Sci Technol. 2015

**Table 2. PFAS Concentrations (ng/L)**

Sample ID	Quantified Using Targeted Analysis	Quantified Using Non-Targeted Analysis				
	GenX	PFMOAA	PFO2HxA	PFO3OA	PFESA BP1	PFESA BP2
Hoffer WTP raw	<LOQ	-	-	-	-	-
Chemours 002	492*	88.9	114	16.0	835	2,970
Bladen Bluffs <sup>a</sup>	<LOQ	-	-	-	-	-
International Paper <sup>a</sup>	49.8	5,050	1,860	249	26.9	642
Brunswick Co. <sup>a</sup>	305*	47.2	72.3	-	52.2	163
CFPUA <sup>a</sup>	121	236	630	421	84.1	1,450
Pender Co. <sup>a</sup>	54.8	4,800	2,730	438	41.4	679
L&D #3 CFR	45.2	70.6	73.1	7.96	216	196

<sup>a</sup> Samples from finished drinking water sites.

**Flag Codes:**

<LOQ	Less than limit of quantitation (LOQ)
*	Analyte above upper range of calibration curve (250 ng/L), consider as estimate.
- (dash)	Analyte not detected